LANSCE DIVISION RESEARCH REVIEW

SMARTS—Providing New Capabilities in Materials Research

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Introduction

Residual stress in fabricated components can significantly augment or detract from their service lifetimes. Accordingly, measurement of its presence helps determine whether its effect will be beneficial or detrimental. Therein lies part of the motivation behind the construction of SMARTS (Spectrometer for MAterials Research at Temperature and Stress). SMARTS is a third-generation neutron diffractometer that will enter commissioning in June 2001 and will provide an exciting range of capabilities for studying polycrystalline materials. Its focus includes measurement of spatially resolved strain-fields, phase deformation and load transfer in composites, the evolution of stress during temperature (or pressure) fabrication, and the development of strain during reactions (such as reduction, oxidation, or other phase transformations). The technique that underpins the instrument is neutron diffraction, which has been used to study engineering structural materials since the early 1980s.

SMARTS will expand the use of neutron diffraction to a wider range of engineering problems than is currently possible. With its extensive array of *in situ* capabilities for sample environments, it will enable measurements on small (1 mm^3) or large (1 m^3) samples. Ease of access to the sample position is one significant new feature. Components with dimensions up to 1 m and up to 1500 kg can be positioned precisely in the beam. Permanently mounted alignment theodolites (located several meters from the specimen) will provide a simple and efficient way to position samples or equipment to within 0.01 mm. Achieving this level of precision is critical for stress-strain measurements because misalignments of more than 0.1 mm can result in significant pseudo-strain artifacts.

A furnace and load frame suite will allow research on materials under extreme loads (40,000 lb) and at extreme temperatures (1500°C). *In situ* uni-axial loading on samples up to 1 cm in diameter at stresses of 2 GPa under vacuum or in a controlled atmosphere will be routine. These values represent a significant increase over what is currently achievable.

Research Opportunities with SMARTS

Fabricated components. Residual stress in materials is often a life-determining feature because it can contribute to the aging processes by increasing susceptibility to crack formation. Interest in fabricated components crosses institutional boundaries with relevance to industry, academia, and the defense community. SMARTS will be able to accommodate large, heavy, hazardous, and radioactive samples.

New materials. Materials that could be studied on SMARTS range from cemented carbide composites for tool bits to continuous-fiber-reinforced, titanium-matrix alloys for the aerospace industry. The unique characterization capability will allow the examination of materials developed for both mundane and esoteric purposes. One particular focus will be validation of deformation models that describe microstructural effects associated with texture.

Ceramics research. One area of research that is likely to be fruitful concerns ceramics—specifically, in the study of spatial and temporal progression of solid-state reactions and phase transformations. The high-temperature, mechanical-loading capability of SMARTS will allow, for the first time, investigations of high-temperature structural ceramics and composites under almost real-life conditions.

Spatial resolution capability. Weapons components in the stockpile must be performance-ready for many years beyond their original design life. This means that previously ignored residual stresses in critical regions (like welds) may accentuate failure modes that occur during storage and lead to unexpected failure. With SMARTS' spatially resolved capabilities (down to 1 mm^3), experiments can be made on pre-cracked samples perhaps in corrosive environments to monitor crack-tip behavior.

Understanding the implications of remanufacturing. The measurement of residual stress fields is important to the remanufacturing of weapons components. If a new or different fabrication process is used, a component may experience a different residual-stress state than its predecessor. The result may be cracking or distortion of a system that was hitherto stable. SMARTS will aid researchers by measuring the strain state of remanufactured components, which is critical to their final shape and strength, their *on-the-shelf* performance, and ultimately to their performance in a weapon system.

Process monitoring. SMARTS offers *in situ* monitoring to follow in real time powder processing, sintering, welding, mechanical alloying and design, and stress-relief procedures. Future inclusion of an environmental chamber will enable studies of *in situ* hydrogen embrittlement (HEM) and HEM-assisted crack growth in uranium and uranium-niobium alloys.

What Distinguishes SMARTS from the Neutron Powder Diffractometer?

The SMARTS mode of operation is similar to that of the current spectrometers NPD (Neutron Powder Diffractometer) and HIPD (High-Intensity Powder Diffractometer) or of the future HIPPO (High-Pressure Preferred Orientation) diffractometer. However, because the existing engineering research program takes place on the NPD, it is the most appropriate instrument for comparison. What distinguishes SMARTS from NPD is the use of a neutron guide, a T_0 chopper, a large accessible sample area, and a six-fold increase in detector coverage.

Neutrons pass from a chilled water moderator through a series of scrapers (in the bulk shield) to the entrance of the neutron guide in ER-1. A break in the guide at 10 m from the moderator accommodates a T_0 chopper and provides space for a future frame-definition chopper. The T_0 chopper removes fast neutrons and the gamma flash (to minimize background) while the frame-definition chopper will be required if the source repetition rate is increased to 30 Hz. After the T_0 chopper, slow thermal neutrons pass down the guide to the entrance of the cave. The guide terminates approximately 3 m from the sample (at the inner surface of the cave wall). Two aperture sets (located between the exit of the guide and the sample) permit the beam cross section to be defined continuously in shape and area between 1 and 100 mm^2.

Samples will be placed on a translator that can accommodate up to 1500 kg, move in three orthogonal directions, and rotate about a vertical axis. A wide range of ancillary equipment can be accommodated on the translator, including cryostats, furnaces, texture goniometers, high-pressure cells, and load frames (Fig. 1a). Access through the roof of the cave allows large objects to be lowered into the sample position using the 15-ton (ER-2) crane. Under extreme cases, the downstream wall can be removed without affecting the integrity of the remaining cave to allow fork-lift access to the sample position. For measurements requiring spatial resolution, one of five different radial collimators (each offering different characteristics) can be supported from the roof

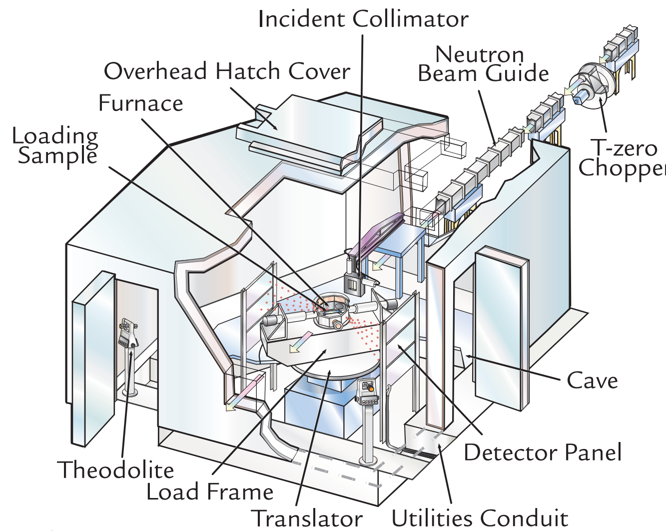


Fig. 1a

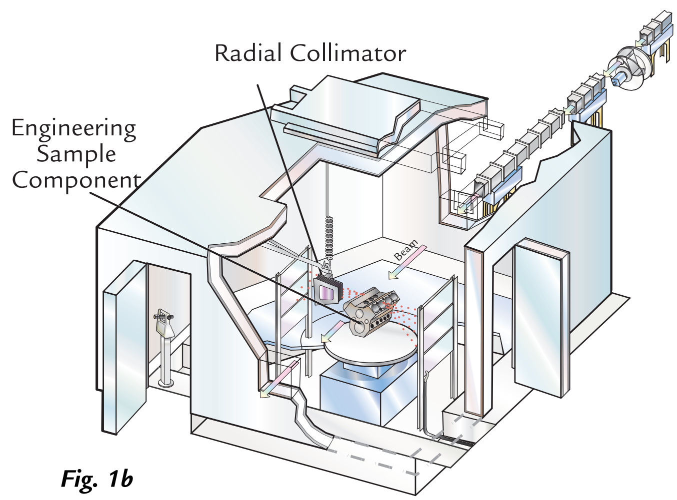


Fig. 1b

▲ ***Fig. 1.*** *Neutrons from the moderator pass through a series of collimating apertures before entering the neutron guide. In ER-1, a break in the guide accommodates a T-zero (T_0) chopper, which removes fast neutrons and gamma flash that would otherwise contribute unwanted background. Slow thermal neutrons continue down the guide to the entrance of the SMARTS cave. On exiting the guide, neutrons pass to the center of the cave where some are scattered by the sample to the detectors. Samples or ancillary systems are placed directly on the translator, which can accommodate up to 1500 kg, move in three orthogonal directions, and rotate about a vertical axis. Theodolites provide a precise optical triangulation and alignment capability for equipment or samples. Fig. 1a (cutaway of the cave) illustrates the load-frame-furnace suite in position. Note that there is no collimation between the sample and the detector. Fig. 1b (cutaway of the cave) shows a radial collimator between the detector and a generic engineering sample. When used with the incident collimation, selection of an appropriate radial collimator defines a sampling volume for spatially resolved measurements.*

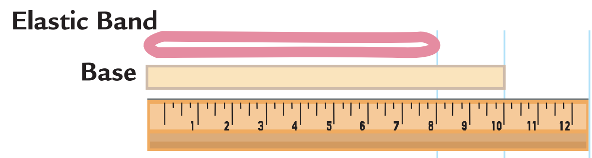
between the sample and the detector as needed (Fig. 1b).

When the beam penetrates a sample, a small fraction of the neutrons interact with atoms in the material and scatter in all directions. Some are scattered to two detector banks centered on the horizontal plane at 90° to the incident beam. Each detector comprises three panels with a total of 192 ^{3}He gas-filled tubes. The panels are tilted relative to the incident beam to balance the resolution from the low to high scattering angles. The tilt compensates for the change in resolution caused by the angular placement of each panel with a corresponding change in the sample-to-detector flight path that varies with 2θ. Interactions between the neutrons and ^{3}He in the detector tubes produce ^{4}He plus gamma radiation and ionize the gas, creating a cascade of electrons with associated charges. These charges are digitized and converted electronically to patterns of intensity versus scattering angle. Data from the tubes will be combined to provide time-of-flight (TOF) neutron-diffraction patterns. Analysis of the diffraction patterns will use Rietveld codes, such as the GSAS (Generalized Structures Analysis System). Data acquisition will be based on VME (virtual memory extension) technology and will use Web-based visualization and control software. Experiments can be controlled remotely from the user's laboratory, and real-time data analysis will use a software package called SMARTS-EXPERT.

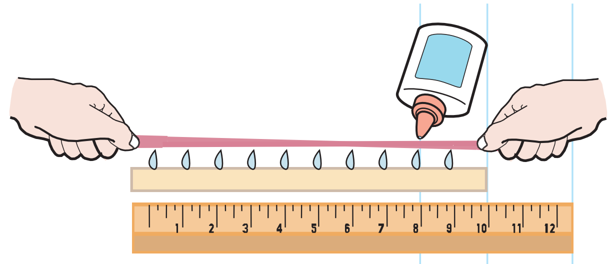
Residual Stresses in Manufactured Objects

When a material experiences a force (often expressed as a stress, which is force per unit area), the result is twofold. Most obvious is a distortion in shape that may or may not be visually obvious. Less obvious is a change in the amount of subsequent stress that can be applied before failure. The change can be either closer to or further from a state of failure. Any stress, no matter how small, produces these results. In the case of a stiff material (or a small stress), the change may be small, but it will always be present. What makes this significant is that the same effect can be produced in the absence of an applied stress by what is termed a residual stress.

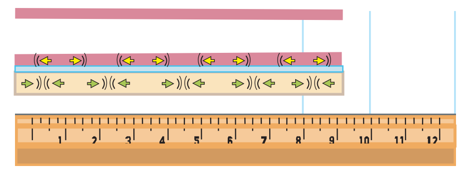
Residual stresses are *locked into* a material in the absence of a stress. As an analogy, consider a stretched elastic band, which is then glued to a card (Fig. 2). After the glue is dry, the *stretching* force that holds the elastic band is removed. Despite the removal of this force and some limited contraction, the band remains taut because the glue holds it to the card. Accordingly, the resistance to stop the elastic band from contracting comes from the card; this resistance results in a state of residual stress between the card and the band. A qualitatively similar effect can be produced in manufactured components. (There is also stored energy in the system, which can be released if a crack propagates through the residual stress field.)



Take an elastic band that, in its relaxed state, is 8" long and a flat base that is 10" long.



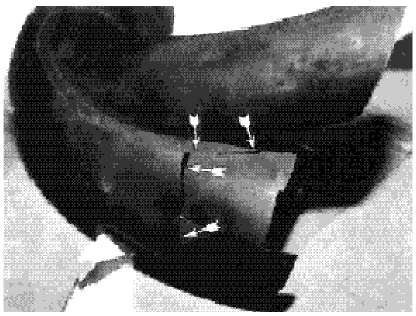
Stretch the elastic band so that its length matches the base. Then while holding it taut, glue it to the base.



After the glue dries, let go of the elastic band, which will try to contract but can't because it is glued to the base.

▲***Fig. 2.*** *Residual-stress-origin analogy. Resistance to the elastic band's attempt to recover to its relaxed state come from the base, which compresses. The net effect leaves the elastic band in a state of tension (⇦⇨) and the base in a state of compression (⇨⇦). In a component, the effects that lead to residual stress are fabrication processes such as machining or welding–processes that deform one region with respect to another. The gross deformities are often invisible to the eye but may be associated with a stress that leaves the component close to failure. Also distortion may occur during subsequent operations (such as machining or heat treatment) as residual stress is redistributed.*

In manufactured materials, the processing implicit in fabrication more often than not introduces residual stresses. Inhomogeneous heating and cooling, phase transformations during heat treatment, and localized plasticity during machining are all processes that can introduce residual stresses. In extreme cases, these processes can substantially alter the strength available for an application. Thus durability, fatigue, fracture toughness, and strength are all affected. Consequently, the ability to measure (and predict) the presence of residual stress is a critical technology for material processing, stress relief, heat treatment, lifetime prediction, and alloy design. Because a residual stress is locked inside an object, it may only become apparent long after fabrication, perhaps when a failure occurs at a significantly lower stress than anticipated resulting from a design that did not account for it (as shown in Fig. 3). In metallic objects where residual stresses may be a fraction of a millimeter or many centimeters from a free surface, a measuring tool that probes the interior is needed. Because they have no electric charge, neutrons penetrate many centimeters into most materials (with less attenuation than x-rays), making them ideal for examining, in a *nondestructive* manner, the interior of polycrystalline materials. The value of neutrons can be readily seen in their application to critical components, such as welds in pressurized-water-reactor vessels or in NASA rocket boosters.



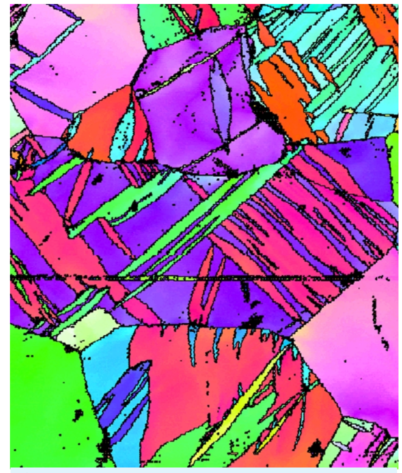
▲ ***Fig. 3.*** *Example of residual stress. This aircraft manifold cracked along a welded seam and across the dent in one of the pipes (arrows).*

SMARTS—Understanding Deformation at the Microstructural Level

In most engineering calculations, the mechanical performance of structures or components are calculated under the assumption that the material is homogeneous. Although this assumption is often sufficient, it may become dramatically invalid under atypical conditions such as heat or pressure or close to failure.

Polycrystalline materials (that comprise a large fraction of the materials used in structural engineering applications) are comprised of many individual grains (Fig 4). These grains typically exhibit elastic and plastic characteristics that are directionally anisotropic. Elastic moduli are often anisotropic, and plasticity occurs through slip along preferentially oriented crystallographic directions. The net result is that deformation at the microstructural level is far from homogeneous!

Neutron diffraction provides a technique to study the influence of the polycrystalline structure on the strain response of individual lattice planes. Because neutron measurements can irradiate bulk volumes (as much as several cm^3), the results are statistically robust and representative of the bulk. Moreover, the ability to pass the beam through heat shields or vacuum containment simplifies performing *in situ* deformation studies under extreme conditions.



▲ ***Fig. 4.*** *False color microstructure showing grains and twins in zirconium (courtesy of George Kaschner, MST-Division).*

The response of different lattice planes under applied loads can be predicted with micro-mechanics models that either describe microstructures as discrete entities or as *average* agglomerates. For instance, self-consistent elasto-plastic polycrystalline models have predicted residual strains associated with defined texture states. The predictions were validated by *in situ* neutron diffraction measurements under load, which provided a rigorous test of the applicability and limitations of the model. However, despite some successes, models ultimately need to predict the onset and gradual evolution of intergranular strains associated with plastic cold-working. In this respect, the effects of initial texture and dislocation structure have been poorly studied, and in particular current models fare poorly when simulating large strain problems such as rolling. For this reason, neutron measurements are important since they can test the validity of modeling assumptions concerning grain reorientation (i.e., texture development) and the hardening evolution in individual grains.

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